12:1. The acylation step is desirably carried out at a mildly elevated temperature, such as from 40 to 75°C, and often at from 45 to 60°C. The reaction is advantageously monitored via HPLC and/or proton NMR and allowed to continue until hepta-acylated cellobiose is no longer detectable. Such reaction period is often in the range of from 10 to 30 hours. The acylation product can be recovered by cooling to ambient and precipitation in methanol. This method is particular applicable for the preparation of esters predominantly in the β anomeric form.

In the second step in the above-mentioned three step process, the acylated cellobiose produced in the first step or the variant, i.e. in predominantly α or β anomeric form or a mixture thereof, is partially de-acylated preferentially at the anomeric carbon. One method comprises reacting the fully acylated cellobiose with a mixture of a low molecular weight aliphatic acid, (C1-C4) and especially acetic acid with an alkylene diamine such as in particular ethylene diamine, at a low concentration in THF (tetrahydrofuran), such as from 4 to 15% by weight acylated cellobiose. The acid employed in the second step has a higher pK_a than the catalyst in the first step. The reaction preferably employs an approximately equimolar ratio of acid to acylated cellobiose, such as in the range of 0.9 to 1.2:1 and a small molar excess of diamine to acylated cellobiose, such as from 1.6 to 2.5:1. The reaction can conveniently be carried out in at or about ambient temperature, e.g. 20 to 30°C for a long reaction time, often of at least 12 hours and particularly from 24 to 60 hours, or at a mildly elevated

temperature such as up to 45°C for a commensurately shorter period such as selected in the range of from 5 to 10 hours. The resultant partially deacylated material can be recovered by extraction into a haloalkane solvent such as dichloromethane and acid washed. After drying, it is recrystallisable from a THF/methanol mixture.

In the third step, the partially de-acylated cellobiose is re-acylated. The re-acylation can employ a carboxylic acid, an acid chloride, or an anhydride.

In the first variant of this third step, the cellobiose is reacted with an at least equimolar amount of an acid chloride of formula R'COCl, preferably a small molar excess of from 1.1 to 1.5:1, in the presence of at least an equimolar amount of triethylamine and preferably a small molar excess of from 1.1 to 1.5:1. The reaction is desirably conducted at or within 10°C of reflux temperature, suitably for at least 1 hour and preferably from 2 to 4 hours. Desirably, the reaction is permitted to continue until at least a predetermined extent of acylation at the anomeric carbon, such as at least 80% and often at least 90% has occurred. In this first variant, the resultant CHME is predominantly in the β anomeric form, the exact proportions of the α and β forms depending on the acyl substituent and process conditions adopted.

In the second variant of this third step, the partially de-acylated cellobiose is reacted with an substantial excess of a carboxylic acid of formula $R'CO_2H$, such as a mole ratio of at least 50:1 and particularly from 60 to 100:1 in the

presence of a significant molar excess of a strong acid catalyst such as that employed in the first step and preferably in a mole ratio to the cellobiose of at least 20:1 and especially from 22:1 to 50:1. The reaction is preferably carried out at elevated temperature, such as especially above 90°C and particularly at about 100°C. The reaction period is desirably at least 4 hours and is especially from 5 to 10 hours. Desirably, the reaction is permitted to continue until at least a predetermined extent of acylation at the anomeric carbon, such as at least 80% and often at least 90% has occurred. In this second variant, the resultant CHME is predominantly in the α anomeric form, the exact proportions of the α and β forms depending on the acyl substituent and process conditions adopted.

In the third variant for carrying out the third step, the partially-deacylated cellobiose is reacted with an anhydride of formula $(R'CO)_2O$. The reaction is conveniently carried out in a hydrocarbon solvent having a boiling point of at least 80°C, such as toluene. The reaction preferably employs an excess anhydride, especially in a mole ratio to the cellobiose of at least 2:1, and often from 2.5 to 10:1.

Water-immiscible liquid

The water-immiscible liquid, which in many embodiments acts as a carrier for a disperse solid or liquid phase, normally comprises one or a mixture of materials which are relatively hydrophobic so as to be immiscible in water. Some hydrophilic liquid may be included in the water-immiscible